DIONEX

LIQUID CHROMATOGRAPHY MODULE-3
OPERATOR'S MANUAL

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SECTION 1
INTRODUCTION

1.1 GENERAL

The Liquid Chromatography Module (P/N 043075), or LCM-3, is an integral part of the Dionex DX-300 Gradient Chromatography Systems. The LCM-3 houses a Rheodyne 9126 injection valve, a column select valve, separator and guard columns, suppressor devices, a detector cell, and all interconnecting tubing. The Rheodyne 9126 injection valve and the column select valve are controlled externally by valves 5 and 6 of the Advanced Gradient Pump (AGP). The LCM-3 may also contain an optional column heater (P/N 035246) and an optional Thermal Stabilizer (P/N 043117).

1.2 SPECIFICATIONS

1.2.1 General

- Dimensions: 17.25 cm H x 43.75 cm W x 47.5 cm D (6.9 in x 17.5 in x 19 in)
- Weight: 37.4 kg (17 lbs)
- Temperature Requirements: Ambient temperature range 10° to 40°C (50° to 104°F)
- Operating Humidity Range: 5-95% relative humidity (non-condensing)
- Air Pressure Requirements: 60-100 psi for the Rheodyne 9126 injection valve; 100-120 psi for the column select valve. Use laboratory-quality air or regulated compressed air, nitrogen, or helium.
- Operating Pressure: 5000 psi (340 atm.), maximum

1.2.2 Hydraulics

- Rheodyne 9126 Injection Valve: 5000 psi (340 atm.) metal-free rotary valve for low-volume injections.
- Column Select Valve: 4000 psi metal-free slider valve which can be used to direct eluent and sample to one of two columns.
  The column select valve is installed at the factory, but is not plumbed into the system. The tubing required to plumb the LCM-3 for column selection applications is included in the LCM-3 Ship Kit (P/N 044699).
- The length of all interconnecting tubing installed in the module should minimize void volume in the system.

1.2.3 Column Heater (Optional)

- Power Rating: 300 W
- Materials: Anodized aluminum
- Operating Temperature: 30, 40, 50, 60, or 70°C
- Dimensions: 3 cm H x 27 cm L x 5 cm W (1.25 in x 10.5 in x 2 in)

SECTION 2
DESCRIPTION

2.1 HYDRAULIC DESIGN

Eluent from the Advanced Gradient Pump enters the Rheodyne 9126 injection valve port inside the Liquid Chromatography Module. From the injection valve, eluent and sample flow through the guard column, separator column, suppressor device or other post- column device (e.g., a Membrane Reactor), and finally through the detector cell(s).

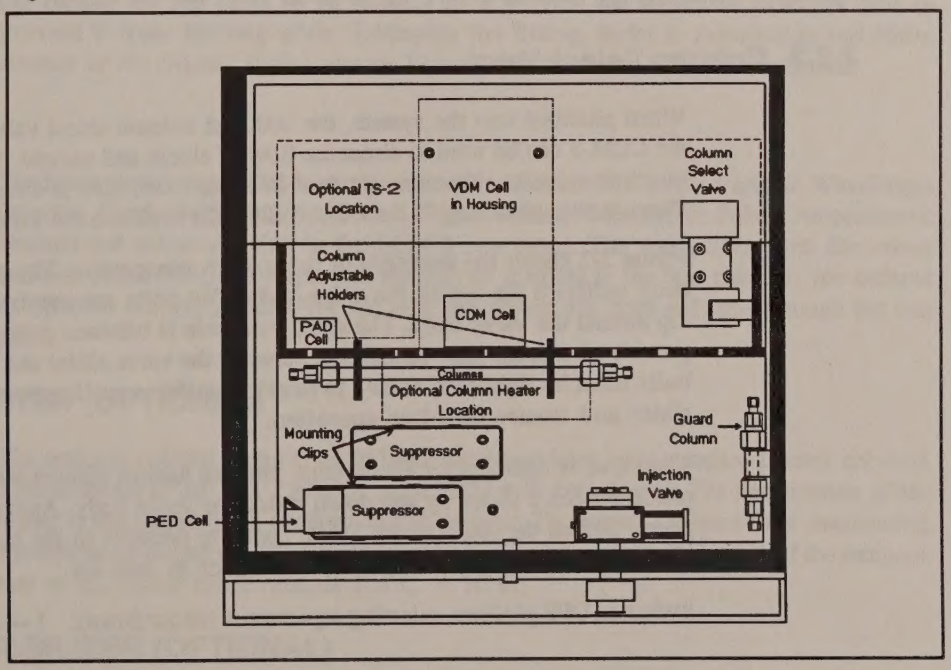
The injection valve is controlled externally by valve 5 of the Advanced Gradient Pump. When valve 5 is off, the valve is in the LOAD position and sample is loaded into the sample loop. When valve 5 is on, the injection valve is switched to the INJECT position. This places the sample loop in the flow path, injecting sample into the eluent stream, and onto the top of the separator column.

Although the LCM-3 includes a column select valve, the valve is not usually plumbed into the system. However, there is enough tubing in the LCM-3 Ship Kit (P/N 044699) to plumb the valve and a second column into the system. The column select valve is controlled externally by valve 6 of the Advanced Gradient Pump.

2.2 INTERIOR OF THE LCM-3

Figure 2-1 shows the location of the components inside the LCM-3.

Figure 2 - 1
Interior of the LCM-3
(Top View)



- The Rheodyne 9126 injection valve is mounted on the inside of the front panel door. The bulkhead fitting to the left of the valve provides an alternate method of filling the sample loop (see Section 4.1 for additional information).
- The column select valve is mounted in the rear right corner, behind the center mounting panel.

- The MicroMembrane™ suppressors are mounted on the left front of the module floor. They are held in place by spring mounting clips.
- The separator columns are mounted in holders on the front of the center mounting panel. Retaining bars hold the columns in place.
- The guard columns are mounted in retainers on the right-hand wall.
- The cells for the Conductivity Detector Module and Pulsed Amperometric Detector are mounted on the rear of the center mounting panel. The Pulsed Electrochemical Detector cell is mounted behind the front panel, in the left corner. The Variable Wavelength Detector cell is installed in the housing in the rear of the module.

2.3 VALVES

2.3.1 Injection Valve

The Rheodyne 9126 injection valve is a low-volume, metal-free, 5000 psi rotary injection valve. The valve accommodates syringe injections or pressurized sample loading. The LCM-3 Ship Kit includes the following accessories for the Rheodyne 9126 injection valve: a 25 µL gas-tight syringe (P/N 041389) and a 25 µL sample loop (P/N 042857). For more information about the valve, including important operating precautions, refer to your *Rheodyne Valve Operator's Manual* (Document No. 034468), included in the LCM-3 Ship Kit.

2.3.2 Column Select Valve

When plumbed into the system, the 4000 psi column select valve in the rear section of the LCM-3 can be used to direct the flow of eluent and sample from the Rheodyne 9126 injection valve to either column A or B. From there, flow is directed to the detector cell. There is enough tubing in the LCM-3 Ship Kit to plumb the valve and a second column.

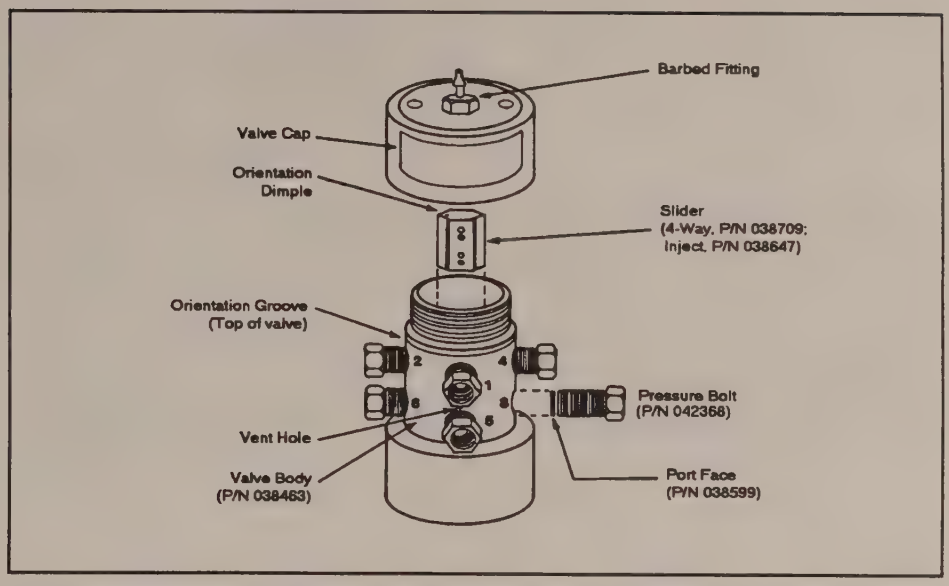
Figure 2-2 shows the assembly of the column select valve. The groove around the valve body indicates the top of the valve body. The ports are numbered clockwise from the top around the valve body. The small vent hole is between ports 1 and 5. Pressure bolts provide an intermediate connection between the valve slider and all tube fittings. **These bolts must be tightened evenly to provide uniform sealing pressure against the valve slider and ensure leak-free operation.**

The valve is operated by air pressure pushing against pistons inside the valve cap. The piston pushes a slider up and down inside the valve body. Applying pressure to the top piston pushes the slider down, while applying pressure to the bottom piston pushes the slider up. The slider connects the valve ports as follows:

Switch in OFF position, selecting separator A (slider down): 1 → 4 and 2 → 3
5 → 9 and 6 → 7

Switch in ON position, selecting separator B (slider up): 1 → 2 and 3 → 4
5 → 6 and 7 → 8

Figure 2 - 2
Column Select Valve



The valve is tested to operate leak-free at 4000 psi. After testing, the valve pressure bolts are loosened to reduce stress on critical parts during storage. Before shipment, the pressure bolts are tightened to provide leak-free operation at pressures up to 2500 psi (90 in-oz torque). **If you need to operate at higher pressures (up to 4000 psi), first torque the bolts to 128 in-oz. If the valve is unused for more than two days, reduce the torque on the bolts to 90 in-oz. Use a wrench on the valve pressure bolt to prevent it from turning while tightening the fitting.** Refer to *Installation and Maintenance of the Dionex High-Pressure Valve* (Document No. 032678) for details.

2.4 REAR PANEL

The cables inside the LCM-3 which connect the detector cell to the Variable Wavelength Detector, Conductivity Detector, Pulsed Electrochemical Detector, or Pulsed Amperometric Detector exit via an opening in the LCM-3 rear panel. The waste lines from the valves and cell inside the LCM-3 also exit through an opening in the rear panel. If you ordered an optional column heater (P/N 035246), the heater line cord will exit through the rear panel.

2.5 COLUMN HEATER (OPTIONAL)

The optional column heater (P/N 035246) accommodates most standard Dionex columns. Temperatures of 30°, 40°, 50°, 60°, and 70°C may be selected. The temperature of the heater is monitored by an encapsulated thermistor. To prevent columns from overheating, a temperature-sensitive safety switch automatically turns off the AC power if the temperature of the heater block exceeds 105°C ± 10°C.

2.6 THERMAL STABILIZER (OPTIONAL)

If desired, the detector cell can be installed inside an optional Thermal Stabilizer, or TS-2, (P/N 043117). The Thermal Stabilizer improves baseline stability by damping fluctuations in ambient temperature, thus improving detection at trace levels.

SECTION 3
INSTALLATION

3.1 UNPACKING AND INSPECTION

Inspect the Liquid Chromatography Module (LCM-3) for shipping damage as soon as you receive it. Many carriers limit their claim period to 10 days after delivery, so report any shipping damage to the incoming carrier immediately.

The Ship Kit (P/N 044699) contains all the parts needed to install the LCM-3. The contents of the Ship Kit are listed on the packing list. Check the contents of each package against the packing list to verify that you have received all components. To request help in assessing shipping damage or to report a missing or damaged part, contact your nearest Dionex Regional Office.

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3.2 FACILITIES REQUIRED

3.2.1 Pneumatic Pressure

CAUTION: To avoid contamination and possible deterioration of the Advanced Gradient Pump, make sure laboratory air, if used, is oil-free, dry, and filtered.

3.2.1.1 Valves

The pneumatic cylinders of the Rheodyne 9126 injection valve are operated by the two air solenoid valves for valve 5 of the Advanced Gradient Pump (AGP). Use a regulated source of laboratory-quality nitrogen, helium, or air pressure to provide the necessary 60-120 psi.

The column select valve is operated by the two air solenoid valves for valve 6 of the AGP. If the valve is plumbed into the system, use a regulated source of laboratory-quality nitrogen, helium, or air pressure to provide the 80-120 psi required for the valve.

3.2.1.2 Eluent Reservoirs

All eluents must be degassed, filtered, and stored in pressurized reservoirs. This helps prevent bubbles from forming in the eluent selector valves and assures optimal operation of the Advanced Gradient Pump. Refer to Sections 1.4.1 and 1.4.1.1 of the *Advanced Gradient Pump Operators Manual* (Document No. 034463) for details.

The eluent reservoirs require a regulated source of helium pressure for degassing eluents and pressurizing reservoirs. The pressure applied to the reservoirs must be regulated to 7 ± 2 psi. Dionex recommends installing an Eluent Degas Module (P/N 037124) to provide continuous, on-line helium sparge-degassing of eluents and keep the reservoirs pressurized with helium. Use the Eluent Degas Module or install a regulator (P/N 038201) on the supply line to the reservoirs to reduce the pressure to a maximum of 7 + 2 psi. The *Eluent Degas Module Manual* (Document No. 034042) contains complete installation instructions.

Use only helium to sparge-degas eluents; other gases are too soluble in most liquids. If helium is unavailable, pressurize the reservoirs with argon or nitrogen. Vacuum-degas eluents daily if either of these gases is used to pressurize the reservoirs.

3.2.1.3 Regenerant/Reagent Reservoirs

The MicroMembrane Suppressor regenerant requires a pressurized reservoir or an Auto-Regen Accessory. The Membrane Reactor reagent requires a pressurized reservoir. Use a cylinder of nitrogen or other inert gas with a regulator (P/N 038201) to provide the required pressure (5 psi for the regenerant, 20 psi for the reagent). Refer to the manual included with each of these for complete installation instructions.

3.3 INSTALLATION INSTRUCTIONS

If any difficulties arise during installation, contact the nearest Dionex Regional Office (Section 3.1).

3.3.1 Waste Line Installation

Install the piece of 12.5-mm (0.5-in) OD tubing (P/N 035759) over the waste nipple in the lower right corner of the LCM-3 rear panel. Make sure the end of the line is below the level of the LCM-3 and that it is not elevated at any point.

CAUTION: Neutralize acidic and caustic waste before disposal. Do not dispose of wastes containing organic solvents through the municipal sewage system.

3.3.2 Air Connections to the Advanced Gradient Pump

Valves 5 and 6 of the AGP are each controlled by two air solenoid valves. The solenoid valves are mounted in a vertical column on the left wall of the AGP, in the following order: valve 5, top; valve 5, bottom; valve 6, top; and valve 6, bottom. See Figure 4-7 of the *Advanced Gradient Pump Operators Manual* (Document No. 034463).

The table below summarizes the function of each solenoid and the color of tubing connected to it. Refer to this table when making air connections between the AGP and LCM-3.

Solenoid	Position	Function	Tubing Color
Valve 5, top	OFF	Selects LOAD	Orange
Valve 5, bottom	ON	Selects INJECT	Yellow
Valve 6, top	OFF	Selects separator A	Green
Valve 6, bottom	ON	Selects separator B	Blue

1. Locate the yellow air tubing connected to the bottom solenoid for valve 5. Using a barbed union (P/N 035529), connect the tubing to the yellow air tubing exiting the LCM-3 rear panel.

2. Locate the orange air tubing connected to the top solenoid for valve 5. Using a barbed union (P/N 035529), connect the tubing to the orange air tubing exiting the LCM-3 rear panel.

3. For column select applications, locate the green tubing connected to the bottom solenoid for valve 6. Using a barbed union (P/N 035529), connect this tubing to the green air tubing exiting the rear of the LCM-3.

4. Using a barbed union (P/N 035529), connect the blue tubing from the bottom solenoid for valve 6 to the blue tubing exiting the LCM-3 rear panel.

5. After making all the required air connections, turn on the air supply and adjust the pressure to 100-120 psi. Actuate valve 5 several times to make sure there are no air leaks. (A short burst of air is released when a valve is actuated; a continuous hissing indicates a leak.) Refer to Section 5.3.4 if you hear an air leak.

   To turn on valve 5, press **% 5 1** on the AGP front panel once, press **STOP/START** to turn off the valve, press **% 5 0** once, and then press **STOP/START** twice.

6. To plumb the LCM-3 for column select applications, repeat Step 5 for valve 6 of the AGP. To turn valve 6 on and off, press **% 6 1** and **% 6 0**.

3.3.3 Liquid Line Connections

3.3.3.1 System Tubing and Fittings

The LCM-3 Ship Kit contains PEEK tubing in three different IDs. After selecting the appropriate tubing size (see below), you will cut the tubing to the lengths required to plumb the system. However, before cutting any tubing, be sure that it is long enough to allow you to service the system (e.g., you should be able to open the front door of the LCM-3 and pull the drawer out of the enclosure).

- For 4-mm column systems, use 0.25-mm (0.010-in) ID PEEK tubing (P/N 042690) for connections to and from system components. Use 0.5-mm (0.020-in) ID PEEK tubing (P/N 042855) for valve waste lines and 0.25-mm (0.010-in) ID PEEK tubing (P/N 042690) for detector cell waste lines.
- For 2-mm column systems, use 0.125-mm (0.005-in) ID PEEK tubing (P/N 044221) for connections to and from system components. Use 0.5-mm (0.020-in) ID PEEK tubing (P/N 042855) for valve waste lines and 0.25-mm (0.010-in) ID PEEK tubing (P/N 042690) for detector cell waste lines.

The LCM-3 is plumbed with Dionex 10-32 ferrule fittings. If you need instructions on how to install ferrule fittings, refer to *Installation of Dionex Ferrule Fittings* (Document No. 034213), provided in the LCM-3 Ship Kit.

If you prefer, install ThermoFlare tubing where pressures are below 1000 psi; this may include waste lines, tubing from an autosampler to the Rheodyne 9126 valve injection port, and tubing from the separator column to the MicroMembrane Suppressor.

3.3.3.2 Rheodyne Injection Valve Connections

1. Connect the eluent line from the Advanced Gradient Pump pressure transducer to the #2 port on the Rheodyne 9126 injection valve.

2. Connect a piece of PEEK tubing with ferrule fittings to the #3 port on the Rheodyne 9126 valve. Connect the other end of the tubing to the separator column (or guard column, if installed). If necessary, refer to *Installation of Dionex Ferrule Fittings* (Document No. 034213), included in the LCM-3 Ship Kit.

3. Connect the 25 mL sample loop (P/N 042857) between ports #1 and #4 of the Rheodyne 9126 valve.

   NOTE: Other sample loop sizes are available. Contact the nearest Dionex Regional Office for more information.

4. Cut two pieces of tubing for waste lines. Install a ferrule (P/N 043276) and bolt (P/N 043275) on one end of each of the pieces of tubing.

5. Connect the ferrule fitting end of one of the waste lines to the WASTE 1 port on the Rheodyne 9126 valve. Connect the ferrule fitting end of the second waste line to the WASTE 2 port. Direct the waste lines downward through the waste port and place the ends of the lines in a waste container.

3.3.4 Detector Cell Installation

NOTE: When you order a Variable Wavelength Detector (VDM-II) with remote cell option, the detector cell is installed in the LCM-3 at the factory. Follow the instructions in the *Variable Wavelength Detector-II Operators Manual* (Document No. 032955) to connect the cables from the detector to the cell.

When using a detector with a cell **external** to the LCM-3, connect the outlet of the separator, suppressor, or Membrane Reactor to the detector cell inlet (see Figure 3-1). Route the tubing exiting the separator, suppressor, or Membrane Reactor through one of the slots at either side of the LCM-3 front panel and over to the VDM-II.

Follow the instructions below to install a Variable Wavelength Detector, Conductivity Detector, Pulsed Amperometric Detector, or Pulsed Electrochemical Detector cell **inside** the LCM-3. Refer to the plumbing schematics in Figures 3-2, 3-3, 3-4, or 3-5 when installing a cell inside the LCM-3. If the cell is installed inside the optional Thermal Stabilizer (TS-2), also see Section 3.3.5.

1. Carefully pull the LCM-3 forward, out of the enclosure, and place it on the workbench. Use a Phillips screwdriver to loosen the four screws holding the top cover in place.

2. Connect the cell inlet into the desired position in the flow path.

3. Using the screw (P/N 037624) and wing nut (P/N 030495) provided in the Ship Kit, mount the cell on the rear of the mounting panel in the center of the LCM-3 (Figure 2-1). Orient the cell so that the inlet is at the bottom and the outlet is at the top (flow should be from bottom to top). This helps prevent bubbles from being trapped in the cell.

   NOTE: As an alternative, the PAD cell may be mounted on the left side of the LCM-3 mounting panel. To do so, locate the mounting bracket (P/N 036259) provided in the PAD Ship Kit. Mount the bracket vertically on the left rear side of the support panel, using the screw (P/N 037624) and wing nut (P/N 030495) provided in the LCM-3 Ship Kit. Attach the PAD cell to the bracket by pushing the cell body onto the projecting studs.

4. Connect a piece of tubing to the exit of the CDM, PED, PAD, or VDM cell. Direct the end of this tubing either to waste or to additional valves and/or columns.

5. Insert the cell cables, one connector at a time, through the grommeted opening in the center of the rear panel. Extend the cell cables from the LCM-3 and attach the cables to the detector as described in the detector manual.

6. Slide the module back into the enclosure.

Figure 3 - 1
Plumbing Schematic for a Detector Cell Outside the LCM-3

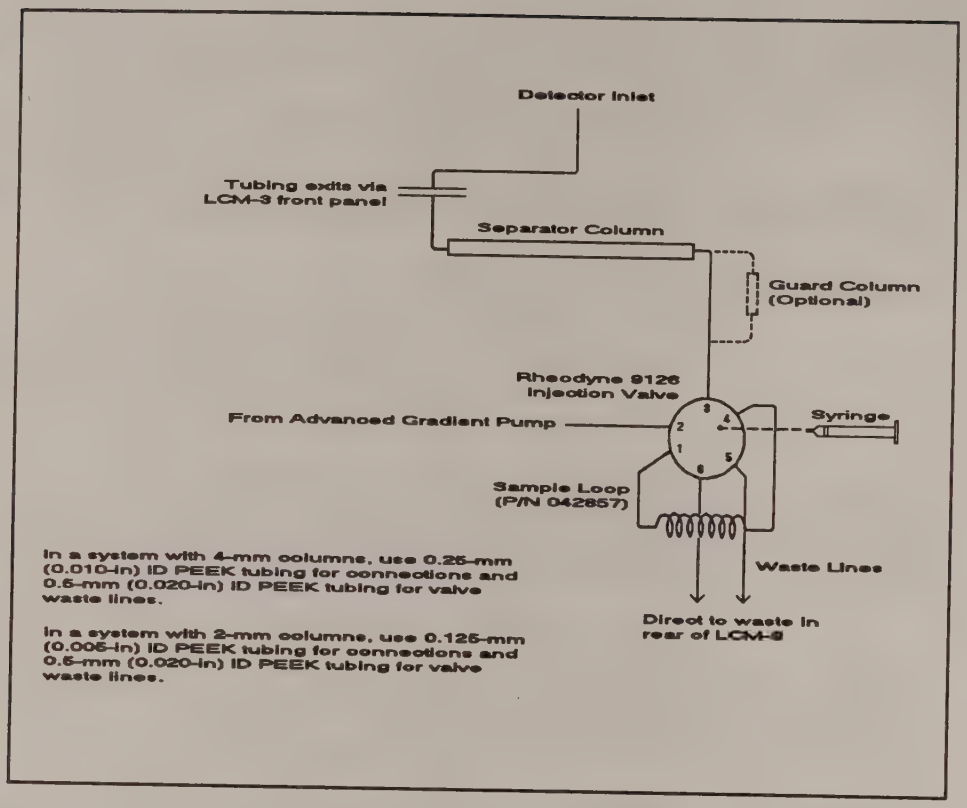

Detector Inlet
Tubing exits via LCM-3 front panel
Separator Column
Guard Column (Optional)
Rheodyne 9126 Injection Valve
From Advanced Gradient Pump
Syringe
Sample Loop (P/N 042857)
Waste Lines
Direct to waste in rear of LCM-3
In a system with 4-mm columns, use 0.25-mm (0.010-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
In a system with 2-mm columns, use 0.125-mm (0.005-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.

Figure 3 - 2
Plumbing Schematic for a VDM Cell

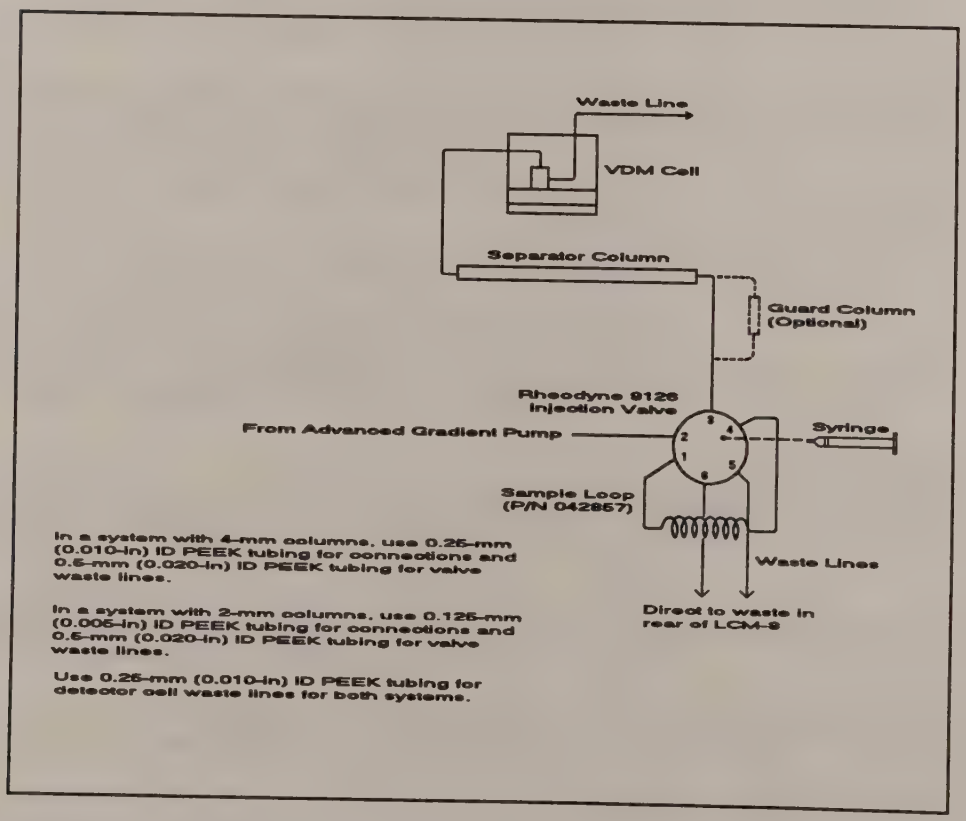

Waste Line
VDM Cell
Separator Column
Guard Column (Optional)
Rheodyne 9126 Injection Valve
From Advanced Gradient Pump
Syringe
Sample Loop (P/N 042857)
Waste Lines
Direct to waste in rear of LCM-3
In a system with 4-mm columns, use 0.25-mm (0.010-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
In a system with 2-mm columns, use 0.125-mm (0.005-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
Use 0.25-mm (0.010-in) ID PEEK tubing for detector cell waste lines for both systems.

Figure 3 - 3
Plumbing Schematic for a CDM Cell

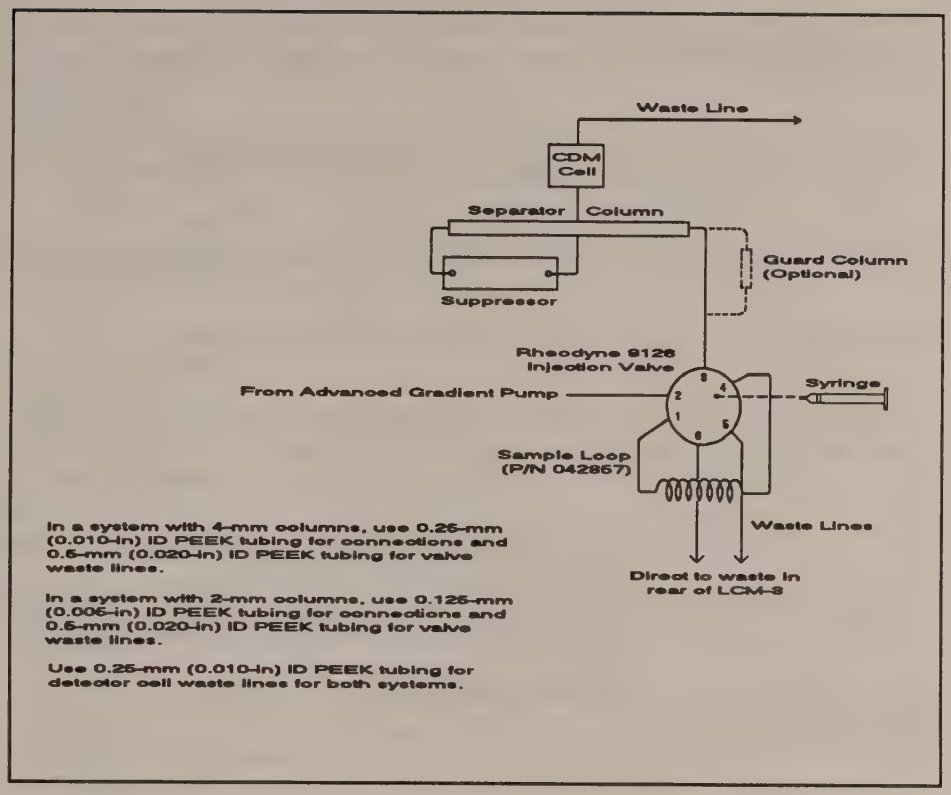

Waste Line
CDM Cell
Separator Column
Guard Column (Optional)
Suppressor
Rheodyne 9126 Injection Valve
From Advanced Gradient Pump
Syringe
Sample Loop (P/N 042867)
Waste Lines
Direct to waste in rear of LCM-3
In a system with 4-mm columns, use 0.25-mm (0.010-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
In a system with 2-mm columns, use 0.125-mm (0.005-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
Use 0.25-mm (0.010-in) ID PEEK tubing for detector cell waste lines for both systems.

Figure 3 - 4
Plumbing Schematic for a PAD Cell

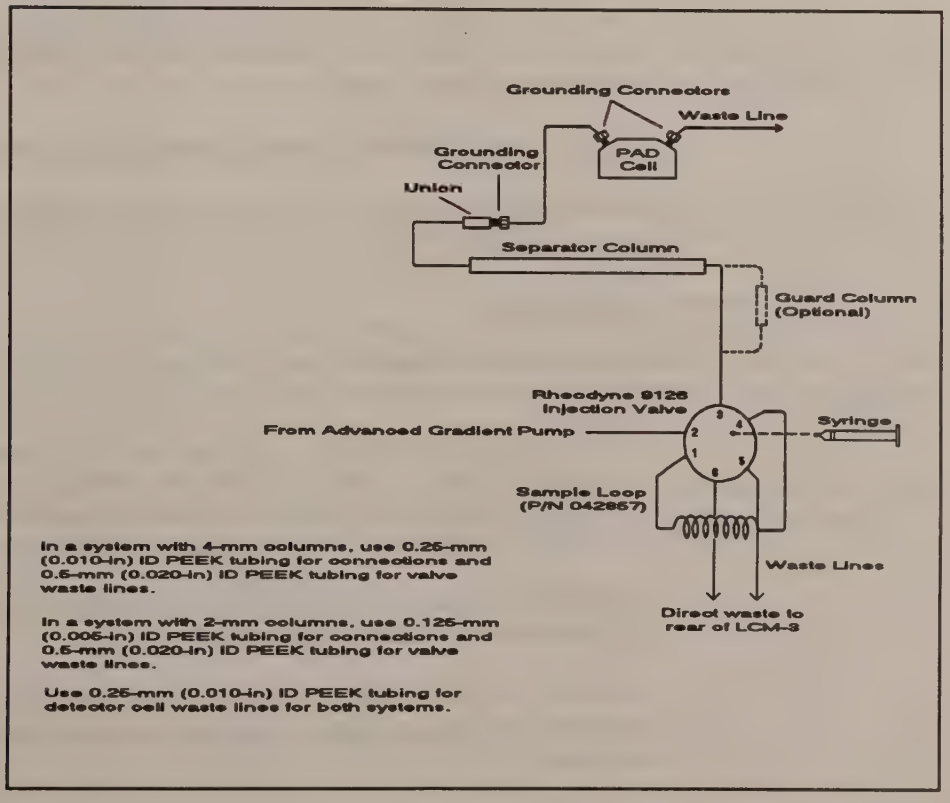
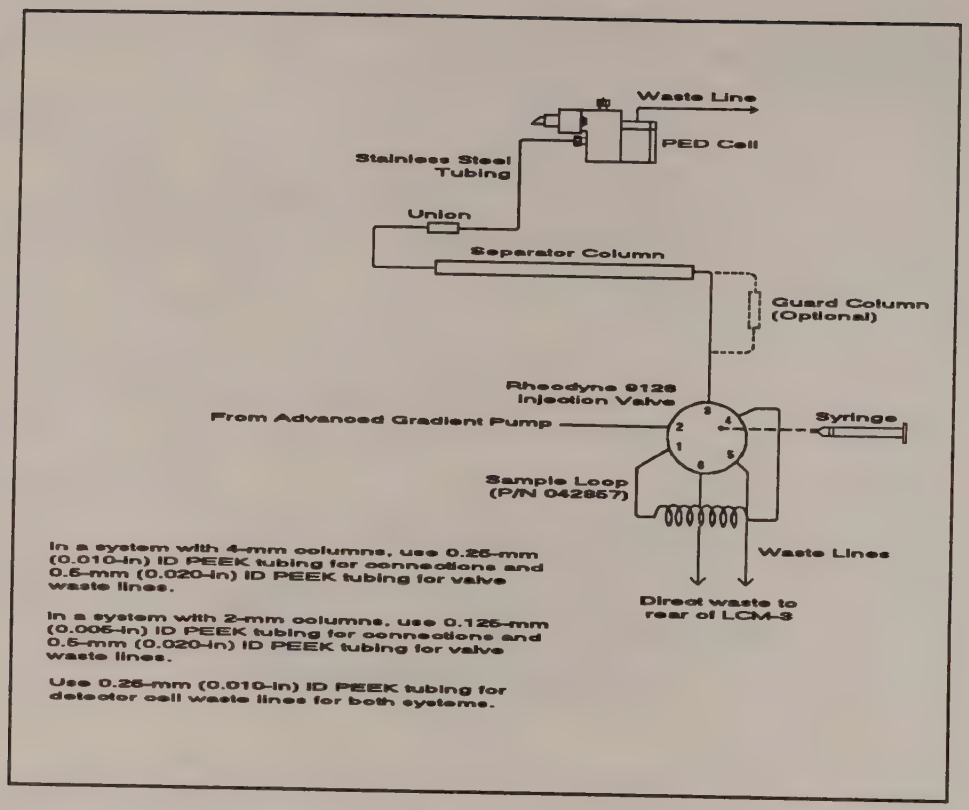

Grounding Connectors
Waste Line
Grounding Connector
PAD Cell
Union
Separator Column
Guard Column (Optional)
Rheodyne 9126 Injection Valve
From Advanced Gradient Pump
Syringe
Sample Loop (P/N 042867)
Waste Lines
Direct waste to rear of LCM-3
In a system with 4-mm columns, use 0.25-mm (0.010-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
In a system with 2-mm columns, use 0.125-mm (0.005-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
Use 0.25-mm (0.010-in) ID PEEK tubing for detector cell waste lines for both systems.

Figure 3 - 5
Plumbing Schematic
for a PED Cell


Waste Line
PED Cell
Stainless Steel Tubing
Union
Separator Column
Guard Column (Optional)
Rheodyne 9128 Injection Valve
From Advanced Gradient Pump
Syringe
Sample Loop (P/N 042857)
Waste Lines
Direct waste to rear of LCM-3
In a system with 4-mm columns, use 0.25-mm (0.010-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
In a system with 2-mm columns, use 0.125-mm (0.005-in) ID PEEK tubing for connections and 0.5-mm (0.020-in) ID PEEK tubing for valve waste lines.
Use 0.25-mm (0.010-in) ID PEEK tubing for detector cell waste lines for both systems.

3.3.5 Thermal Stabilizer Installation (Optional)

1. If the cell is installed in an optional Thermal Stabilizer (TS-2) (P/N 043117), place it on the floor of the LCM- 3, immediately behind the mounting panel in the center of the module (Figure 2-1).

2. Connect the suppressor outlet to the tubing installed in the inlet of the TS-2.

3. Connect a piece of tubing to the outlet of the TS-2. Direct the end of the tubing into a waste container.

3.3.6 Column Installation

NOTE: The separator columns are shipped separately from the Liquid Chromatography Module.

1. Before installing the separator columns, turn on the Advanced Gradient Pump and pump deionized water through the LCM-3 at maximum flow for 1-2 minutes to clear any air from the liquid lines. Activate each valve several times to make sure that no air is trapped in the hydraulic system. Trapped air will reduce efficiency.

2. Reduce the flow rate to 3.0 mL/min. and verify that the pressure through the system, with no columns installed, is less than 100 psi.

3. Refer to the test chromatogram shipped with the column for the flow rate used when the column was tested at Dionex. Select the appropriate flow rate and then turn off the pump.

4. Each column is accompanied by a manual containing specialized installation and start-up instructions. After consulting your column manual for any special requirements, install the columns in the LCM-3:

   a. Remove the end plugs from the separator column and store them in a safe place. You must reinstall the end plugs in the columns before placing the columns in storage.

   b. Use PEEK tubing with ferrule fittings to connect the top of the guard column to the #3 port of the Rheodyne 9126 valve.

   c. For conductivity detection, connect the bottom of the separator to the eluent inlet of the suppressor device.

   d. Connect the eluent outlet of the suppressor device to the detector cell. Place the suppressor on the stand- offs on the floor of the drawer and rotate the small retainers into place over the suppressor.

3.3.7 Connecting Regenerant to the Suppressor

1. Assemble and fill the regenerant reservoir as described in the instructions shipped with the reservoir.

2. Locate the 75-cm (30-in) tubing assembly (P/N 035727) provided in the MicroMembrane Suppressor Ship Kit. Use this tubing to connect the reservoir to the REGENERANT INLET of the suppressor.

3. Direct the line connected to the suppressor REGENERANT OUTLET to waste. Adjust the regenerant flow rate as instructed in the suppressor manual.

4. Place the suppressor on the floor of the module. Loosen the retainer thumbscrews and rotate the retainers into place over the suppressor. Tighten the thumbscrews.

3.3.8 Column Heater Installation (Optional)

1. Remove the screws holding the halves of the column block together. Separate the halves and insert the column. Reassemble the block, leaving the screws projecting slightly more than 0.625 cm (0.25 in).

2. Remove the column holders from their slots in the center mounting panel of the LCM-3. Note the two narrow slots in the panel. Mount the column heater on the panel by sliding the screws through to the bottom of the narrow slots and then tightening the screws by hand.

3. Route the column heater line cord through the wide slot to the right of the heater, through the rear section of the LCM-3, and plug it into the system power strip.

SECTION 4
OPERATION

4.1 ROUTINE OPERATION

During an analysis, eluent from the Advanced Gradient Pump enters the flow path through the LCM-3 front panel and goes directly to the Rheodyne 9126 injection valve. There are four methods of filling the Rheodyne valve sample loop:

- *Direct injection into the valve*. Insert the syringe into the needle port on the Rheodyne valve.
- *Sucking from the waste port*. Insert the syringe into the needle port on the Rheodyne valve and draw sample into the loop through the injection valve waste line (port #6). No injected sample will come into contact with the metal needle of the loading syringe.
- *Loading from the waste port*. Connect a luer fitting and syringe to the bulkhead fitting (to the left of the Rheodyne valve) on the LCM-3 front panel. Connect the Rheodyne valve waste line to the rear of this bulkhead fitting. Insert the needle adaptor shipped with the Rheodyne valve into the needle port. (If necessary, refer to the *Rheodyne Valve Operating Instructions* included in the LCM-3 Ship Kit.) Push sample through the waste line. Waste will exit the valve through the needle adaptor. No injected sample will come into contact with the metal needle of the loading syringe.
- *Loading from the Automated Sampler (ASM)*. Connect the ASM sample valve to port #6 (waste) of the Rheodyne valve, using some of the PEEK tubing and ferrule fittings provided in the LCM-3 Ship Kit. (Before selecting the tubing size, see the **NOTE** below.) Route the PEEK tubing from the ASM sample valve, through the **SAMPLE IN** fitting on the ASM front panel, through whichever clearance slot in the LCM-3 door is nearest the ASM, and finally to port #6 of the Rheodyne injection valve. Insert the needle adaptor shipped with the Rheodyne valve into the needle port. Connect the 180-cm (72-in) long ThermoFlare tubing assembly (P/N 041019) to the needle adaptor. This 0.925-mm (0.037-in) ID tubing is the valve waste line. Trim the free end of the tubing so that it is just long enough to reach the waste container.

  NOTE: The LCM-3 Ship Kit contains PEEK tubing in two sizes: 0.25-mm (0.010-in) (P/N 042690) and 0.125-mm (0.005-in) ID (P/N 044221). Use the 0.25-mm (0.010-in) ID tubing for the sample line. Keep the sample line as short as possible to ensure that the backpressure in the sample delivery circuit remains below 690 kPag (100 psi), which is the maximum the ASM can deliver sample against. When calculating tubing length, include the line from the sample vial to the sample valve in the ASM (a 60-cm (24-in) long piece of 0.3-mm (0.012-in) ID tubing), the line connecting the ASM and LCM-3, and the waste line exiting the valve. The tubing length also depends on the TYPE selected at the ASM front panel. When the selected TYPE is LOOP, the maximum tubing length is 109 cm (25 in). When the selected TYPE is CONC, the maximum tubing length is 434 cm (120 in).

After injection, eluent and sample flow through the guard column, separator column, suppressor device or other post-column device (if used), and finally through the detector cell(s).

NOTE: There are two methods of filling the Rheodyne 9126 injection valve sample loop: complete-filling and partial-filling. These methods differ in accuracy, precision, and the amount of sample required. Read Sections 8.1 and 8.2 of the *Rheodyne Valve Operating Instructions for the Model 9125* before selecting a filling method for your application.

4.2 COLUMN HEATER OPERATION

Turn on the POWER switch and set the TEMP knob to the desired temperature.

4.3 SHUTDOWN

If the LCM-3 will be unused for more than two days, reduce the torque on the pressure bolts on the column select valve (if the valve has been in use) to 90 in-oz to prevent possible damage to the valve.

SECTION 5
MAINTENANCE AND SERVICE

5.1 ROUTINE MAINTENANCE

The following maintenance procedures are especially important for keeping the Liquid Chromatography Module operating reliably:

- Periodically check for leaks or spills inside the drawer. Locate and repair leaks (Section 5.2.1) and clean up spills. Rinse any dried eluents or reagents off system components with deionized water or, in the case of spilled ninhydrin, with isopropyl alcohol.
- Periodically check all air and liquid lines for crimping. Relocate pinched lines and replace damaged lines.

5.2 TROUBLESHOOTING GUIDE

Use the Troubleshooting Guide to isolate and solve problems that can occur while operating the LCM-3. If there is more than one possible cause, read over the potential sources of the problem to determine which may be the most applicable. If the problem persists, call the nearest Dionex Regional Service Office (see Section 3.1).

For troubleshooting procedures specific to the Rheodyne 9126 injection valve, refer to the *Rheodyne Valve Operators Manual* (Document No. 034468).

5.2.1 Liquid Leaks

Possible Cause	Possible Remedy
A. Leaking fitting.	Make sure all liquid line connections are tight: 1. If the connections are made with Dionex ferrule fittings, see *Installation of Dionex Ferrule Fittings* (Document No. 034213) for instructions. 2. If the column select valve is plumbed into the system, remove the valve from its mounting clip to improve access to all valve ports. **Use a wrench on the valve pressure bolt to prevent it from turning while tightening the fitting.**
B. Broken liquid line.	Cut the tubing at the break and replace the ferrule (Section 5.3.1.1) or replace the tubing.
C. Blocked or improperly installed waste line.	Make sure the waste lines from the valves and cells are not crimped or otherwise blocked. Also make sure they are not elevated at any point after they exit the rear of the module.

5.2.1 Liquid Leaks (continued)

Possible Cause	Possible Remedy
D. Pressure bolt(s) on column switching valve are too loose.	Follow the procedure below to tighten the pressure bolts: 1. Turn off the liquid flow. 2. Tighten each pressure bolt fingertight, then use an open end wrench to tighten an additional one-eighth turn. **NOTE:** For optimum leak-free operation, all eight pressure bolts must be evenly tightened against the slider. 3. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear. **NOTE:** For optimum leak-free operation, a torque wrench may be used to evenly tighten pressure bolts. Refer to *Installation and Maintenance of the Dionex Inert High-Pressure Valve* (Document No. 032678) for torque requirements.
E. A column select valve port face is scratched.	Replace the port face (Section 5.3.6).

5.2.2 Excessive System Backpressure

Possible Cause	Possible Remedy
A. Restriction in the hydraulic system	Check all liquid lines and valves for crimping or blockage (Section 5.3.2). Make sure the ferrule fittings are not overtightened where Tefzel tubing is used. If necessary, refer to *Installation of Dionex Ferrule Fittings* (Document No. 034213) for details.
B. Flow rate through the columns is too high.	Reduce the flow rate.
C. Clogged column bed supports.	Replace the bed supports as instructed in the column manual.
D. Columns are contaminated.	Clean the columns (see *Column Rejuvenation Procedures*, Technical Note 2R, Document No. 032036).
E. A blockage in the membrane suppressor or a contaminated suppressor membrane.	Refer to the membrane suppressor manual for troubleshooting procedures.

5.2.3 Inoperative Column Select Valve

Possible Cause	Possible Remedy
A. Air is not reaching valve.	Check that the air supply is turned on and is supplying 80-120 psi.
B. Air tubing harness is crimped or pinched.	Check the air tubing for blockage. Remove and replace any blocked tubing (Section 5.3.2). Reposition crimped or pinched tubes.
C. Air leak. Air leaks are usually audible.	Isolate and eliminate leaks (Section 5.3.4).
D. Valve is incorrectly plumbed.	Check that the system is correctly plumbed.
E. Pressure bolt(s) on the column select valve are too tight.	Follow the procedure below to loosen the pressure bolts if the column select valve is plumbed into the system: 1. Turn off the liquid flow. 2. Loosen each pressure bolt slightly. 3. Tighten each pressure bolt fingertight, then use an open end wrench to tighten an additional one-eighth turn. **NOTE:** For optimum leak-free operation, all eight pressure bolts must be evenly tightened against the slider. 4. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear. **NOTE:** For optimum leak-free operation, a torque wrench may be used to evenly tighten pressure bolts. Refer to *Installation and Maintenance of the Dionex Inert High-Pressure Valve* (Document No. 032678) for torque requirements.

5.2.4 Air Leaks from Column Select Valve

Possible Cause	Possible Remedy
A. Air leaks are usually audible and frequently cause excessive air consumption.	Locate and repair the leak (Section 5.3.4).

5.2.5 Peak Ghosting

Ghosting is the appearance of extraneous peaks in a chromatogram. These may be late-eluting peaks from a previous injection or may result from a contaminated valve or a poor sample loading technique. These peaks may co-elute with peaks of interest, resulting in non-reproducible peak heights.

Possible Cause	Possible Remedy
A. Insufficient time between sample injections.	Wait until the previous sample has been completely eluted before making another injection.
B. Insufficient flush between samples.	Flush sample loop with at least 10 loop volumes of deionized water or sample between sample injections.

5.2.6 Non-Reproducible Peak Height and/or Retention Time

Possible Cause	Possible Remedy
A. Insufficient time between sample injections.	Wait until the previous sample has been completely eluted before making another injection.
B. Insufficient flush between samples.	Flush the sample loop with at least 10 loop volumes of deionized water or sample between sample injections.
C. Column overloading.	1. Change to a sample loop with a smaller volume. 2. Dilute the sample.
D. Liquid leaks.	Refer to Section 5.3.3 to locate and eliminate leaks.

5.2.7 Abnormal Retention Time and/or Selectivity

Possible Cause	Possible Remedy
A. System is not equilibrated following an eluent change.	Allow the system to equilibrate with at least 20 column volumes of eluent (for example, 30 minutes at 3.0 mL/min. for 4-mm anion separator columns, 0.75 mL/min. for 2-mm anion separator columns, or 2 hours at 0.7 mL/min. for ICE separator columns) before beginning an analysis.
B. Flow rate through system is incorrect.	1. Check that the correct flow rate is selected. 2. Locate and eliminate any liquid leaks (Section 5.3.3).
C. Contaminated or incorrect eluent.	Remake the eluent, using reagent grade chemicals and ASTM filtered Type I (18 megohm or 1 µS) grade deionized water.
D. Contaminated or degraded sample.	Take appropriate precautions when preparing and storing samples to prevent contamination and degradation.
E. Column is contaminated.	1. Clean the column as described in the column manual 2. If cleaning is unsuccessful, replace the column.

5.3 SERVICE

This section describes service and repair procedures for the LCM-3. These are not required very often, but some wear of the fittings and tubing is to be expected during the life of the module. Before repairing or replacing any part, consult Section 5.2 to isolate the source of the problem. For service and maintenance procedures for the Rheodyne 9126 valve, refer to the Rheodyne manual supplied in the Ship Kit.

When ordering replacement parts, please include the model and serial number of your LCM-3, along with the part numbers and, where applicable, the revision number of the items you are ordering.

5.3.1 Tube Fitting Installation

The LCM-3 is plumbed with Dionex 10-32 ferrule fittings throughout the system. PEEK tubing is recommended for use throughout the system. However, ThermoFlare tubing may be installed where pressures are below 1000 psi; this may include waste lines, tubing from an autosampler to the Rheodyne 9126 valve injection port, and tubing from the separator column to the MicroMembrane Suppressor. Use 1.6-mm (0.063-in) ID Teflon tubing for regenerant lines for both the 2-mm and 4-mm MicroMembrane Suppressors.

For instructions on how to install ferrule fittings on PEEK or Tefzel tubing, refer to *Installation of Dionex Ferrule Fittings* (Document No. 034213), provided in the LCM-3 Ship Kit.

5.3.2 Isolating and Eliminating a Restriction in the Hydraulic System

A restriction in the hydraulic system will cause excessive system backpressure.

1. Begin pumping eluent through the system (including the columns) at the flow rate normally used.

2. Following the appropriate hydraulic schematic (Section 3.3.4), work backward through the system, beginning at the cell exit. One at a time, loosen each fitting and observe the pressure. The connection at which the pressure drops indicates the point of restriction.

3. Remove the restriction either by flushing or by replacing the section of tubing.

4. If the restriction is caused by the column select valve, disassemble the valve and clean the slider (Section 5.3.5). If cleaning the valve slider does not eliminate the backpressure problem, replace the port face (Section 5.3.6).

5.3.3 Isolating and Eliminating Column Select Valve Liquid Leaks

A poor seal between the ferrule sealing surface and the valve pressure bolt or between the port face (Figure 2-2) and the valve slider may cause the column select valve to leak. The flow rate through the system may be low, producing chromatograms with longer than normal retention times and reproducibility may be poor.

1. Tighten any loose fittings fingertight, then an additional one-eighth turn. Tighten further only if a leak continues. **DO NOT OVERTIGHTEN!** Use an open end wrench to prevent valve pressure bolts from turning when tightening connections to the valves.

2. If tightening the fitting does not stop the leak, disconnect the fitting from the valve. Occasionally, fittings stop leaking if simply disconnected and then immediately reconnected. If the leak continues, install a short length of tubing, with fittings, between the pressure bolt and the suspect fitting. Connect the fitting to the tubing with a union. A leaking fitting will also leak inside the union. If necessary, replace the fitting. **If you replace, the fitting, also replace the ferrule.**

   NOTE: When connecting or disconnecting fittings from the valve pressure bolts, use a wrench to prevent the nut from turning.

3. Make sure all valve pressure bolts are evenly tightened. Refer to *Installation and Maintenance of the Dionex Inert High-Pressure Valve* (Document No. 032678) for the torque requirements.

   CAUTION: Overtightening may break the bolt off in the valve body.

4. If tightening the pressure bolts does not stop the leak, refer to Section 5.3.6.

5.3.4 Correcting Column Select Valve Air Leaks

An air leak around the valve piston or cap results in excessive air consumption and may cause the valve to operate sluggishly or not at all.

1. Remove the column switching valve from its mounting clip.

2. Make sure that the top and bottom caps are tight. If necessary, tighten them fingertight.

3. If the leak continues, determine which end of the valve leaks by activating the corresponding switch. Air will escape through the vent hole when the leaking end is pressurized.

4. Disconnect the air line from the leaking cap. If necessary, use the valve switch to turn off the air through the disconnected line.

5. Unscrew the leaking cap from the valve body (Figure 2-2). Inspect the cap O-ring for cracks and replace (P/N 038650) if necessary.

6. Press the disconnected air line (Step 4) against the small vent hole between ports 1 and 5. Activate the valve switch to apply air pressure against the slider. The piston will pop out of the valve body.

7. Inspect the piston O-ring and replace it (P/N 038651) if necessary.

8. Carefully lubricate the piston O-ring with a small amount of silicone grease.

   CAUTION: Use the grease sparingly. Excess grease may contaminate the eluent.

9. Press the piston back into the valve body.

10. Make sure that the cap O-ring is inside the cap. Screw the cap back onto the valve body. Tighten it only fingertight.

11. Reconnect the air line to the valve.

5.3.5 Cleaning the Slider in the Column Select Valve

A dirty or plugged valve slider causes excessive system backpressure and may cause poor chromatographic performance.

1. Turn off the pump and the eluent selector valves.

2. Remove the plugged valve from its mounting clip.

3. Disconnect the air line from one cap. If necessary, use the valve switch to turn off the air through the disconnected line.

4. Unscrew one cap from the valve body. Be careful not to lose the cap O-ring.

5. Press the disconnected air line (Step 3) against the small vent hole between ports 1 and 5. Activate the valve switch to apply air pressure against the slider. The piston will pop out of the valve body.

6. Turn off the air supply.

7. Carefully loosen each of the pressure bolts **one-eighth turn** to disengage the port faces from the slider. Do not loosen any further.

8. Remove the other cap. Push on the slider to force the other piston out of the valve body.

9. Carefully push the slider out of the valve body with a blunt rod.

10. Inspect the slider surface. If there are scratches in the surface, replace the slider (P/N 038709).

11. Place the slider in a small container of deionized water or methanol. Sonicate or agitate vigorously for several minutes.

12. Rinse the slider with deionized water. Blow any water out of the holes and inspect them for blockage. Use a fine piece of wire (0.025 in) to dislodge any remaining blockage. Be careful not to scratch the slider surface. If the blockage cannot be removed, replace the slider.

13. Push the slider back into the valve body. Orient the dimple on the top of the slider between ports 1 and 2 of the valve body (Figure 2-2).

    The valve operates properly only if the slider is correctly oriented in the valve body. The groove around the valve body indicates the top of the valve body. The small vent hole is between ports 1 and 5. The ports are numbered clockwise from the top around the valve body. The slider connects the ports as follows:

Switch in OFF or LOAD position (slider down):	1 → 4 and 2 → 3 5 → 8 and 6 → 7
Switch in ON or INJECT position (slider up):	1 → 2 and 3 →4 5 → 6 and 7 →8

14. Inspect the piston O-rings (P/N 038651) and replace them if necessary.

15. Carefully lubricate the piston O-rings with a small amount of silicone grease and reassemble the valve (Section 5.3.4).

16. Tighten each pressure bolt fingertight, then use an open end wrench to tighten an additional one-eighth turn.

    For optimal leak-free operation at 4000 psi, all eight pressure bolts must be evenly tightened against the slider (128 in-oz). A special torque wrench kit (P/N 038943) is available from the Dionex Customer Satisfaction Department.

17. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear.

    CAUTION: Overtightening may break off the bolt in the valve body.

18. If cleaning the valve slider does not eliminate the backpressure problem, see Section 5.3.6.

5.3.6 Replacing a Damaged Port Face (Column Select Valve)

A scratched port face may cause a leak around the pressure bolt. A plugged port face will cause excessive system backpressure and may cause poor chromatographic performance.

1. Turn off the liquid flow.

2. Remove the valve from its mounting clip.

3. Disconnect the tube fitting from the suspect pressure bolt, then unscrew the pressure bolt from the valve body.

4. Inspect the port face. If it is scratched or plugged, carefully pry it out of the pressure bolt, using the extractor tool (P/N 038930) designed for this. Press a new port face (P/N 038599) into the pressure bolt. **DO NOT SCRATCH THE SEALING SURFACES!**

5. Screw the pressure bolt into the valve body. Tighten each pressure bolt fingertight, then use an open end wrench to tighten an additional one-eighth turn.

   For optimal leak-free operation at 4000 psi, all eight pressure bolts must be evenly tightened against the slider (128 in-oz). A special torque wrench kit (P/N 038943) is available from the Dionex Customer Satisfaction Department.

6. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear.

   For optimal leak-free operation, a torque wrench may be used to evenly tighten pressure bolts. Refer to *Installation and Maintenance of the Dionex Inert High-Pressure Valve* (Document No. 032678) for torque requirements.

   CAUTION: Overtightening may break off the bolt in the valve body.

7. Reconnect the tube fitting to the pressure bolt. Use a wrench to prevent the pressure bolt from turning while tightening the fitting. Tighten the fitting fingertight, then an additional one-eighth turn. Tighten further only if leaks appear.

READER'S COMMENTS FORM

Liquid Chromatography Module-3 Operator's Manual
Document Number 034520, Release 02

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